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Agency

Prevention, Pesticides
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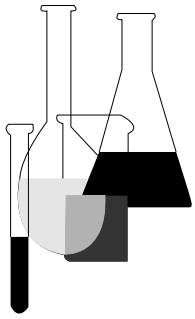
Ecological Effects Test Guidelines

OPPTS 850.7100 Data Reporting for Environmental Chemistry Methods



“Public Draft”

INTRODUCTION

This guideline is one of a series of test guidelines that have been developed by the Office of Prevention, Pesticides and Toxic Substances, United States Environmental Protection Agency for use in the testing of pesticides and toxic substances, and the development of test data that must be submitted to the Agency for review under Federal regulations.

The Office of Prevention, Pesticides and Toxic Substances (OPPTS) has developed this guideline through a process of harmonization that blended the testing guidance and requirements that existed in the Office of Pollution Prevention and Toxics (OPPT) and appeared in Title 40, Chapter I, Subchapter R of the Code of Federal Regulations (CFR), the Office of Pesticide Programs (OPP) which appeared in publications of the National Technical Information Service (NTIS) and the guidelines published by the Organization for Economic Cooperation and Development (OECD).

The purpose of harmonizing these guidelines into a single set of OPPTS guidelines is to minimize variations among the testing procedures that must be performed to meet the data requirements of the U. S. Environmental Protection Agency under the Toxic Substances Control Act (15 U.S.C. 2601) and the Federal Insecticide, Fungicide and Rodenticide Act (7 U.S.C. 136, *et seq.*).

Public Draft Access Information: This draft guideline is part of a series of related harmonized guidelines that need to be considered as a unit. *For copies:* These guidelines are available electronically from the EPA Public Access Gopher (gopher.epa.gov) under the heading ‘‘Environmental Test Methods and Guidelines’’ or in paper by contacting the OPP Public Docket at (703) 305–5805 or by e-mail: guidelines@epamail.epa.gov.

To Submit Comments: Interested persons are invited to submit comments. By mail: Public Docket and Freedom of Information Section, Office of Pesticide Programs, Field Operations Division (7506C), Environmental Protection Agency, 401 M St. SW., Washington, DC 20460. In person: bring to: Rm. 1132, Crystal Mall #2, 1921 Jefferson Davis Highway, Arlington, VA. Comments may also be submitted electronically by sending electronic mail (e-mail) to: guidelines@epamail.epa.gov.

Final Guideline Release: This guideline is available from the U.S. Government Printing Office, Washington, DC 20402 on *The Federal Bulletin Board*. By modem dial 202–512–1387, telnet and ftp: fedbbs.access.gpo.gov (IP 162.140.64.19), or call 202–512–0135 for disks or paper copies. This guideline is also available electronically in ASCII and PDF (portable document format) from the EPA Public Access Gopher (gopher.epa.gov) under the heading ‘‘Environmental Test Methods and Guidelines.’’

OPPTS 850.7100 Data reporting for environmental chemistry methods.

(a) **Scope**—(1) **Applicability.** This guideline is intended to meet testing requirements of the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) (7 U.S.C. 136, *et seq.*) and the Toxic Substances Control Act (TSCA) (15 U.S.C. 2601).

(2) [Reserved]

(b) **Data reporting.** (1) Registrants must provide EPA with accurate and precise data on the performance of those soil and water chemistry methods that are used to develop laboratory and/or field residue data to support exposure, environmental fate, and ecological effects studies for registration and reregistration. This includes all single or multianalyte methods for the parent compounds, toxicologically significant metabolites, and degradates in each environmental matrix. Registrants shall provide EPA with clearly written and complete analytical methods that describe the exact procedure, materials, and equipment to be used by regulatory chemists to validate their methods. Analytical methods shall be practical, rapid, and quantitate analytes of interest. The Agency will accept best available methods for those parent compounds, toxicologically significant metabolites, and degradates that have significant risks and require state-of-the-art equipment to measure trace amounts of analytes. Both practical and rapid and best available methods must use equipment that is commercially available in the United States. If methods use conventional gas chromatography, liquid chromatography, etc., the registrants shall submit confirmatory methods using GC/MS, LC/MS, second column or other suitable procedures. GC/MS and LC/MS methods that are used as the primary method to generate study data do not require another confirmatory procedure. Regulatory chemists should be able to validate practical and rapid analytical methods using a set of samples in twenty-four hours (e.g. three eight-hour working days); however, EPA recognizes that best available methods may require additional time.

(2) EPA regulatory chemists should be able to quickly evaluate soil and water methods for each test level and matrix. Registrants shall submit performance data to EPA demonstrating that an adequate number of samples for each test level were extracted, cleaned up, and analyzed. Each set should have an appropriate number of samples with quality control samples intermingled. This data should support the limit of detection (LOD) or the method detection limit (MDL), the established limit of quantitation (LOQs) and precision and accuracy for each method. The mean recovery at each spiking level, at or above the LOQ, should lie between 70 and 120 percent of the known quantity of the pesticide/metabolite/degradate spiked into the matrix blanks during the method validation. Registrants are to report individual values for recoveries, standard deviations and confidence limits for the pesticide parent, significant metabolites and degradates in fortified samples at each spiking level. The relative standard

deviation (RSD) of replicate measurements of recoveries should not exceed the target level of plus or minus 20 percent at or above the LOQ for each spiking level. EPA recognizes that some methods may not be able to meet these precision requirements. The registrant shall describe how they calculated the values for MDLs or LODs and LOQs and cite any references that they believe are appropriate. Registrants shall not correct sample values for recoveries. Registrants shall also describe any matrix or solvent effects that result in signal enhancement, masking or suppression and the impact those effects have on the test results. Registrants shall clearly identify those laboratories that developed the data for the soil and water methods they submit to EPA. Registrants shall use an independent lab to validate (ILV) soil and water methods that are used to support appropriate exposure, environmental fate and ecological effects studies. A separate report will be prepared by the registrant for the ILV testing and submitted to EPA with the appropriate study. Methods will not be rejected outright for failure to comply with each and every aspect of this DRG but will be reviewed on a case-by-case basis to determine their suitability by science reviewers and chemists in OPP.

(c) **Data reporting format.** The following suggested reporting format is presented to aid the applicant/registrant in generating reports which are compatible with the Agency's review process.

(1) Title/cover page.

(2) Certification. (i) Certification of authenticity by the Study Director (including signature, typed name, title, affiliation, telephone number, and date, etc.).

(ii) Statement of adherence to the FIFRA GLP.

(iii) Statement of claims for non-confidentiality and that the method contains no trade secrets or proprietary data.

(3) Table of contents.

(4) Summary. Provide a brief description of paragraphs (c)(6)(ii) and (c)(6)(iii) of this guideline.

(5) Materials. (i) Equipment (list and describe).

(ii) Reagents and standards (provide MSDS sheets and list and describe the source, purity and stability of analytical grade standards; describe the source and preparation of reagents.

(iii) Safety and Health (describe any special precautions that need to be taken with solvents or reagents and any procedural steps that require special precautions to avoid safety or health hazards).

(6) Methods. (i) Principles of the analytical methods.

(ii) Analytical procedures (describe in a detailed step wise fashion):

(A) Source and characterization of soil samples (e.g. soil (textural class and percent organic matter)) and water samples (pH, turbidity) etc.

(B) Preparation of samples.

(C) Extraction (demonstrate efficiency in specific soil samples from field sites).

(D) Fortifications (i.e. during method validation runs).

(E) Clean-up.

(F) Derivatization (if any).

(iii) Instrumentation. (A) Description (e.g. make/model, type/specifity of detector(s), column(s) (packing materials, size), carrier gas(es), etc.

(B) Operating conditions (e.g. flow rate(s), temperature(s) voltage, etc.).

(C) Calibration procedures (frequency and stability).

(iv) Potential interference(s) (describe the effects of the following on signal enhancement, masking or suppression of signal and their impact on the test results):

(A) Sample matrices.

(B) Other pesticides used on the test site.

(C) Solvents.

(D) Labware.

(v) Confirmatory techniques. Describe confirmatory techniques, i.e. GC/MS, LC/MS, second column, etc.

(vi) Time required for analysis. Give the time required to take a sample/set completely through the analytical procedure, including sample preparation, extraction, cleanup, derivatization, and determination steps. Identify those steps that could significantly increase the time required to complete the method.

(vii) Modifications or potential problems. Specify any allowable variances and describe any unique steps where little variation is allowed in the method. Describe any potential problems and/or modifications that were made to the analytical procedures.

(viii) Methods of calculation. Describe calculations in a step wise fashion and include calibration factors, calibration curves for parent compound, metabolites, and degradates, etc.

(ix) Copies of chromatograms/spectra. Representative sample chromatograms/spectra should be submitted for each analyte measured in each matrix at all spiking levels, including method and matrix blanks. In addition, chromatograms/spectra with the highest levels of background and poorest resolution will also be sent to EPA. Copies of the chromatograms/ spectra are also required for the standards that were used to quantitate the analyte(s) in the representative matrix chromatograms/spectra submitted to EPA.

(x) Other. Describe any and all additional information the registrant considers appropriate and relevant to provide a complete and thorough description of the soil and water method.

(7) Results/discussion. (i) Method validation results (include tables of test levels and results of analysis).

(ii) Accuracy (mean, range of recoveries, standard deviations and confidence limits for specific concentration levels, such as the LOQ or 10X the LOQ).

(iii)) Precision (relative standard deviation at specific concentration levels).

(iv) Limit of detection (provide definitions).

(v) Limit of Quantitation (provide definitions).

(vi) Ruggedness testing (if performed).

(vii) Discussion of selectivity and specificity of method.

(viii) Limitations.

(ix) Independent laboratory validation.

(8) Conclusion. Discuss applicability of the soil and water method for measuring specific test compound(s) in various matrix(ces), ranges, expected recoveries, interference(s), etc.

(9) Tables/figures.

(10) References.

(d) **Independent laboratory validation**—(1) **Guidelines for the registrant.** (i) The registrant should apply the same criteria of quality in selecting an independent laboratory (IL) for environmental chemistry method validations as they would for any other analytical work. An independent lab can be privately or publicly owned or in the registrant's own organization. If that lab is located in the registrant's organization, or is in anyway associated with the development of the original environmental chemistry method (ECM), the same people, equipment, instruments, and supplies (i.e.

glassware, solvents, reagents, standard reference materials) should not be utilized to validate the ECM. The Agency does not expect the registrant to synthesize or purchase another lot of authentic grade analytical standard but it does expect the independent lab to use a new aliquot of that standard in order to prepare spiking solutions for the ILV.

(ii) Furthermore, the personnel conducting the ECM validation should not report to the same study director who was involved in developing the original method or who may have used the method to develop data for field studies to support pesticide registration or reregistration actions. The lab chemists chosen to conduct the independent laboratory validation must be unfamiliar with the method both in its development and subsequent use in analyzing samples collected from field studies. Those individuals, however, should be trained and experienced pesticide residue chemists. The ECM given to the independent lab should be the same one that was used by the registrant to generate field study data. Any significant changes that are made to that method by the chemists at the independent lab should be incorporated into the original method and reported in writing immediately to the registrant. If those changes impact the performance of the original method, such as its precision, accuracy or limit of quantitation, the registrant should report those changes to the Agency.

(iii) Soil and water samples should be identified and/or supplied to the independent lab by the registrant; however, chemists at the independent lab should use those samples to prepare matrix spikes for the validation study. Registrants should select the most difficult matrix to analyze from the appropriate field study identified on the front of this document to demonstrate how the method performs.

(2) **Guidelines for the independent laboratory.** (i) The laboratory conducting the ILV must use the method exactly as it is written by the registrant or another private or public laboratory and should only contact the registrant or developers to clarify minor deficiencies in the method. For example, if the characteristics of the clean up column are not adequately described in the method, the independent laboratory should contact the registrant or developers for clarification. EPA recognizes that chemists in the independent laboratory need to establish the method and that they will need to compute the instrument detection limits and determine the retention times of the analytes.

(ii) They will also need to establish the relationship between the instrument responses and concentrations of analytes and to verify that matrix control samples are free of interferences at the appropriate retention time, wavelength or detector setting. All quality control conditions must be satisfied in order to demonstrate that the method is under control before the IL analyzes any performance samples to be reported to EPA. Any contact with the registrant or developers during the establishment of the method

must be documented in writing in the final report submitted by the independent laboratory.

(iii) The independent laboratory (IL) should be able to quickly evaluate soil and water methods for each test level and matrix. ILs shall submit performance data to registrants demonstrating that an adequate number of samples for each test level were extracted, cleaned up, and analyzed. Each set should have an appropriate number of spiked matrix samples with a method and matrix blank intermingled. This data, and subsequent computations, should support the registrant's limit of detection (LOD) or the method detection limit (MDL), the established limit of quantitation (LOQs) and precision and accuracy for each method. Individual recoveries at each spiking level, at or above the LOQ, should lie between 70 and 120 percent of the known quantity of the pesticide, metabolite and degradate spiked into the matrix blanks during the method validation. Independent labs are to report mean and individual values for recoveries and standard deviations for the pesticide parent, toxicologically significant metabolites and/or degradates in fortified samples at each spiking level. They should also report the confidence intervals (95 or 99%) for the true average recoveries at each spiking level. The relative standard deviation (RSD) of replicate measurements of pesticide concentrations should not exceed the target level of less than or equal to 20 percent for each spiking level at or above the LOQ. EPA recognizes that some methods may not be able to meet these precision requirements. The IL will use the predetermined values from the registrant for LOQs and 10XLOQ to establish appropriate spiking levels. ILs shall not use data from matrix controls (blanks) to correct values from spiked matrix blanks for recoveries. Interferences with peak areas that are less than fifty percent at the MDL/LOD, are considered negligible. ILs shall also describe any matrix or solvent effects that result in signal enhancement, masking or suppression and the impact those effects have on the test results. ILs will prepare a well documented laboratory report to send to the registrant for the ILV and that report with appropriate changes recommended by the registrant will be submitted to EPA with the field study.

(iv) Independent labs will be allowed to analyze three sample sets in order to validate the method as written. A complete set should consist of a reagent blank, two unspiked matrix control samples and five matrix control samples spiked at the LOQ and another five matrix control samples spiked at 10XLOQ for each distinct matrix. A complete set may include more than thirteen samples depending on the number of reagent, unspiked and spiked control matrix samples. It may be necessary, however, to divide a complete set into two subsets for efficient handling. Each subset should contain a reagent blank, two unspiked matrix control samples and five matrix control samples spiked at the LOQ or 10XLOQ.

(v) If the performance data on the first set of samples at any of the required spiking levels is unsuccessful, the independent laboratory may

contact the registrant to clarify the directions given in the method. Any contact with the registrant or developers during the method validation must be documented in writing in the final report submitted by the independent laboratory. If the independent laboratory cannot generate performance data that is similar to the registrant's or developers' after the second set of spiked samples, the independent laboratory may contact the registrant to further clarify the directions given in the method. If the independent laboratory cannot generate performance data that is similar to the registrant's or developers' after the third set, the method should be failed and a report will be sent to the registrant explaining why the method failed. The registrant should then decide whether to repeat the independent laboratory validation at another laboratory, further develop the method or withdraw it.

(vi) A successful ILV will require performance data on at least one complete set of samples that meets those criteria described in paragraphs (d)(2)(iii) and (d)(2)(iv) of this guideline. The revised or rewritten method, performance data, chromatograms and computations from all sets and/or subsets will be sent to the registrant. The registrant, with the assistance of the IL, will decide how to present that information to EPA.